

FORM PTO-1390
(REV 12-29-99)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371

OHSB-285

U.S. APPLICATION NO. (If known, see 37 CFR 1.5)

09/554736

INTERNATIONAL APPLICATION NO
PCT/JP99/00029INTERNATIONAL FILING DATE
08 January 1999PRIORITY DATE CLAIMED
09 January 1998TITLE OF INVENTION ANTISTATIC POLYURETHANE ELASTIC FIBER AND MATERIAL FOR
PRODUCING THE SAME

APPLICANT(S) FOR DO/EO/US

Michihiro SHIBANO, et al.

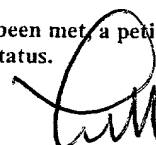
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☐ have not been made and will not be made.
8. ☒ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern document(s) or information included:

1. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☐ A FIRST preliminary amendment.
☐ A SECOND or SUBSEQUENT preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☒ Other items or information:
Copy of PCT/IB/304
Copy of PCT/IB/308

422 Rec'd PCT/PTO 19 MAY 2000

U.S. APPLICATION NO. 609/554736		INTERNATIONAL APPLICATION NO. PCT/JP99/00029		ATTORNEY'S DOCKET NUMBER OHS-285	
17. <input checked="" type="checkbox"/> The following fees are submitted: BASIC NATIONAL FEE (37 CFR 1.492(a)(1)-(5)): Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO \$970.00 International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO \$840.00 International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$690.00 International preliminary examination fee paid to USPTO (37 CFR 1.482) but all claims did not satisfy provisions of PCT Article 33(1)-(4) \$670.00 International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1)-(4) \$96.00 ENTER APPROPRIATE BASIC FEE AMOUNT =				CALCULATIONS <small>PTO USE ONLY</small>	
Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)).				\$	
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total claims	7 - 20 =	0	X \$18.00	\$	
Independent claims	2 - 3 =	0	X \$78.00	\$	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			0	+ \$260.00	\$
TOTAL OF ABOVE CALCULATIONS =				\$ 840.00	
Reduction of 1/2 for filing by small entity, if applicable. A Small Entity Statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28).				\$	
SUBTOTAL =				\$ 840.00	
Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				\$	
TOTAL NATIONAL FEE =				\$ 840.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property				+	\$
TOTAL FEES ENCLOSED =				\$ 840.00	
				Amount to be refunded:	\$
				charged:	\$
a. <input checked="" type="checkbox"/> A check in the amount of \$ <u>840.00</u> to cover the above fees is enclosed. b. <input type="checkbox"/> Please charge my Deposit Account No. _____ in the amount of \$ _____ to cover the above fees. A duplicate copy of this sheet is enclosed. c. <input type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. _____. A duplicate copy of this sheet is enclosed.					
NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.					
SEND ALL CORRESPONDENCE TO: Sherman & Shalloway 413 N. Washington Street Alexandria, VA 22314					
				 SIGNATURE	
				<u>Leonard W. Sherman</u> NAME	
				<u>19636</u> REGISTRATION NUMBER	

BOWERS-001

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the application of:

BOWERS, Donald I.

Serial No.: 09/116,997

Group: 3741

Filed: July 17, 1998

Examiner: E. Swinehart

FOR: MARINE JET DRIVE PUMP PRELOADER FOR REDUCING CAVITATION

Date: May 19, 2000

The Hon. Commissioner of
Patents and Trademarks
Washington, D.C. 20231

AMENDMENT AND REPLY

Sir:

This is in reply to the official action, Paper No. 8,
mailed February 23, 2000, to which a response is due on or before
May 23, 2000.

Please amend the application as follows:

In the Claims

Please amend the claims as follows:

1. (Amended) In a marine jet drive comprising a
housing having a forward intake portion, a rearward discharge
portion and a pump portion disposed therebetween, the pump
portion containing an impeller rotationally driven by a shaft
extending through the intake portion; the improvement comprising
a water propelling device mounted on the shaft forward of the
impeller and within the intake portion of the jet drive housing,
the water propelling device having a diameter which is smaller

than the diameter of the impeller, whereby the water propelling device is rotationally driven concurrently with and at the same speed as the impeller causing water to be moved from the intake portion to the impeller whereby the impeller is preloaded with water across the eye of the impeller thereby reducing the formation of partial vacuum within the water about the impeller whereby cavitation is reduced.

2. (Amended) The marine jet drive of claim 1 wherein the water propelling device is located on the shaft directly in front of the eye of the impeller[, whereby rotation of the water propelling device by the shaft concurrently with the impeller causes water to be moved from the intake portion to the impeller whereby the impeller is preloaded with water across the eye of the impeller thereby reducing the formation of partial vacuum within the water about the impeller whereby cavitation is reduced].

Claim 4, line 1, delete 3"3", insert --1--.

8. (Amended) A method for preloading water in a marine jet drive pump and reducing cavitation in a jet drive pump impeller comprising providing a water propelling device for mounting on a drive shaft of the marine jet drive pump [positioned and] in a position to be rotatable within an intake housing of a marine jet drive unit ahead of and in axial alignment with the eye of the jet drive pump impeller, [the water propelling device drawing] whereby water is drawn from the intake housing into the pump [whereby] thereby preloading the pump with

water across the eye of the impeller, [whereby] thereby reducing the formation of partial vacuum and cavitation within the water about the impeller [is reduced].

9. (Amended) The method of claim 8 [wherein] comprising providing the water propelling device [comprises] in the form of a multibladed propeller [in coaxial alignment with and] having a smaller diameter than the impeller.

10. (Amended) The method of claim 8 [wherein] comprising providing the water propelling device [comprises] in the form of an auger extending along a portion of the shaft ahead of the impeller and within the intake housing of the marine jet drive.

11. (Amended) The method of claim 8 [wherein] comprising providing the water propelling device [comprises] in the form of an axial impeller [in coaxial alignment with and] having a smaller diameter than the pump impeller.

12. (Amended) The method of claim 8 [wherein] comprising providing the water propelling device [has] with a diameter smaller than the impeller of the jet drive pump whereby the periphery of the water propelling device is spaced from the inner surface of the intake housing to permit water flow around the water propelling device.

Please cancel claim 3 in its entirety and without prejudice.

REMARKS

Entry of the foregoing amendments and favorable reconsideration of the present application are respectfully requested in view of the following comments.

Claims 1-21 are currently pending in the application. Claims 13 - 17 have been indicated as being allowed and claims 1, 2, 8-12 and 18-21 have been rejected and claims 3-7 are objected to.

Claims 1, 2, 4 and 8-12 have been amended and claim 3 has been cancelled. Accordingly, claims 1, 2, 4-12 and 18-21 are herewith represented for the Examiner's consideration.

The Examiner has indicated that Claim 13 - 17 are allowable and Applicant takes this opportunity to thank the Examiner for this indication and to accept the allowance of these claims.

The amendments to claims 1, 2 and 4 and the cancellation of claim 3 have been made in response to the Examiner's objection of claims 3-7 and the indication that they would be allowable if rewritten.

Claim 1 has been amended to incorporate the limitation of claim 3 and certain of the material from claim 2. Claim 3 has been cancelled. Concurrently, claim 2 has been amended so as to avoid duplication from claim 1 and claim 4 has been amended to change its dependency from claim 3 to now amended claim 1. Applicant respectfully submits that the amendment of claim 1 in this manner is sufficient to overcome the Examiner's objection

inasmuch as the cited reference fails to teach

"a water propelling device mounted on the shaft forward of the impeller and within the intake portion of the jet drive housing, the water propelling device having a diameter which is smaller than the diameter of the impeller".

Applicant respectfully submits that inclusion of the limitation of claim 2 is not necessary to distinguish over the cited reference and has, therefore, maintained claim 2 as a separate dependent claim.

The amendments to claims 8-12 have been made to present the method of the invention in more proper form and are believed to overcome the Examiner's objection with respect to apparatus limitations in method claims.

Applicant respectfully submits that no new matter has been entered and that the foregoing amendments are properly enterable at this time.

With regard to claims 1,2, 8-12 and 18-21, the Examiner has rejected these claims under 35 U.S.C. 102(b) as anticipated by Chronic, U.S. Patent 4,182,118. The Examiner contends that this reference teaches the claimed invention, including a "preloader" 22 fixed to an upstream portion of the impeller shaft within the intake, for turning at the same rotational speed as the impeller 23. It is the Examiner's contention that the presence of such a preloader will inherently increase water flow as claimed and reduces cavitation, citing the patent at col. 1, lines 53-55.

Applicant respectfully submits that the Examiner has

misread and/or mischaracterized the reference.

Chronic is directed to a jet propulsion engine which uses impeller and stator blade combinations where the blades are optimized for their lifting capacity and which minimizes cavitation in the impeller/stator combination not by means of a preloader but rather by providing an engine housing in the form of a duct which is modified from a theoretical taper so as to be enlarged at each cross-section by an amount substantially equal to the cross-sectional area of the corresponding section through the shafts and blades. This is specified in the reference at Col. 1, line 66 to Col. 2, line 2. Indeed, careful reading of the paragraph at col. 1, lines 53-55, cited by the Examiner, shows that it is the provision of the duct in this form which minimizes cavitation, not the presence of a preloader.

At no point in the reference does Chronic even suggest the inclusion of a preloader within the intake portion of the engine. What the Examiner characterizes as a "preloader", item 22, is clearly identified as an "impeller" by Chronic.

"The drive shaft 18 carries a series of impellers 22, 23 and 24, which are mounted at the entranceway, midway and adjacent the exitway in the duct." Col. 3, lines 4-6.

In addition,

"A series of stators 28, 29 and 30 are disposed alternately between the impeller 22, 23 and 24." Col 3, lines 9-11.

These elements are all contained within the duct which corresponds to the housing 17 and, in fact, become progressively smaller from the first impeller 22 to the third impeller 24 in

view of the taper of the duct. Thus, the structure of Chronic amounts to a multi-stage pump.

The reference fails to disclose or suggest the inclusion of a preloader fixed to an upstream portion of the impeller shaft within the intake, for turning at the same rotational speed as the impeller where the preloader comprises a "a water propelling device mounted on the shaft forward of the impeller and within the intake portion of the jet drive housing, the water propelling device having a diameter which is smaller than the diameter of the impeller". Accordingly, Application respectfully submits that claims 1, 2 and 4-7, as now amended are not anticipated by the cited reference and are, therefore, allowable over the prior art.

As for the method claims 8-12 and 18-21, for the reasons given above, the Chronic reference fails to disclose or suggest the method of the present invention recited in claims 8-12, as amended, involving providing

"a water propelling device for mounting on a drive shaft of the marine jet drive pump in a position to be rotatable within an intake housing of a marine jet drive unit ahead of and in axial alignment with the eye of the jet drive pump impeller".

Nor does the reference disclose or suggest a method as recited in claims 18-21 involving

"increasing water flow from an intake portion of the marine jet drive to the jet drive pump impeller when the watercraft is accelerated, thereby preloading a marine jet drive pump impeller with water from the intake portion of the marine jet drive." (claim 18),

where this is accomplished by,

"providing a water propelling device within the intake portion of the marine jet drive, the device being rotationally driven in combination and simultaneously with the pump impeller." (claim 21)

Chronic reduces cavitation and achieves improved acceleration of water through the jet propulsion engine of the patent by modifying the duct of the housing such that

"the cross-sectional area at any point in the housing 17 is enlarged from an ideal taper by an amount substantially equal to the material thickness of the blades of the respective impellers and stators, the driven shaft and the like." (Col. 3, lines 36-40).

By modifying an ideal taper in this manner, the patent

"... results in a modification of an ideal tube to a constant pressure head producing tube ..." (Col. 5, lines 44-46),

such that

"The pressure head, thus generated, will be relatively uniform throughout the system and internal friction and loss of internal cohesion of the fluid, which results in cavitation, will be minimized." (Col 5, lines 53-57).

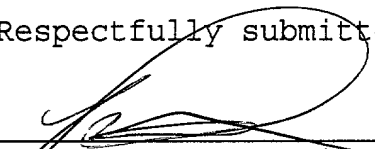
At no point does Chronic discuss or suggest preloading impellers with water drawn from the intake portion of a marine jet drive by providing a water propelling device within the intake portion of the marine jet drive as a method for reducing cavitation and improving the performance of watercraft.

In view of the foregoing, Applicant respectfully submits that the Examiner's rejection of claims 1, 2 and 4-7 in view of the Chronic reference has been overcome, and that the

rejection of claim 8-12 and 18-21 is without support and should be withdrawn since the reference does not teach the present invention.

Accordingly, Applicant respectfully submits that Claims 1, 2, 4-12 and 18-21, are allowable over the prior art, along with claims 13-17, and that the present application is in condition for allowance. An early Notice of Allowance is earnestly solicited.

Respectfully submitted,



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DESCRIPTION

ANTISTATIC POLYURETHANE ELASTIC FIBER AND MATERIAL FOR
PRODUCING THE SAME

5

Technical Field

The present invention relates to an antistatic polyurethane elastic fiber and the material for producing the same.

10

Prior Art

Japanese Patent Publication Laid-Open Hei 7-166426 describes an elastic yarn containing 0.1 to 5 weight percent of sulfonates having C₁₂₋₂₂ hydrocarbon chain, of which surface is coated with a finish containing dimethyl siloxane.

Japanese Patent Publication Laid-Open Hei 1-90258 describes a process for producing antistatic polyurethane foam by dissolving an organic sulfonate and phosphonium salt in a dope for producing polyurethane and by reacting the dope into polyurethane.

Inorganic salts are generated as a byproduct in the production process of sulfonates having hydrocarbon chain, sulfates having hydrocarbon chain and phosphates having hydrocarbon chain. Such metal salts containing the inorganic salts result in fiber breakage and pack choking in fiber extrusion process when they are used in a polymer dope for fiber production, because the inorganic salts are insoluble in polymer dope. Therefore the above-mentioned sulfonates and others must be purified with an organic solvent, such as alcohol, to eliminate the inorganic salts before used in the polymer dope. On the other hand, the above-mentioned sulfonates and others are usually hygroscopic and contain trace of

water. Thus 1 or more weight percent of alcohol and water remain in the above-mentioned sulfonates and others after they are purified into 100 % and treated in vacuum drying. Such sulfonates and others containing
5 alcohol and water lower the degree of polymerization or generate oligomer when they are added in the polymerization process of polyurethane because of the reaction between the alcohol or water in the metal salts and isocyanate. And such polyurethane is spun into fiber
10 of lowered elongation and tenacity.

Disclosure of Invention

The object of the present invention is to provide a material which contains sulfonates and the like having
15 hydrocarbon chain for producing antistatic polyurethane elastic fiber containing the sulfonates and the like having hydrocarbon chain as an antistatic agent.

Another object of the present invention is to provide a material containing minimum alcohol and water
20 to minimize the inhibition of polyurethane formation due to the reaction between the alcohol or water and isocyanate for the purpose of producing antistatic polyurethane.

Further object of the present invention is to provide a material, in which sulfonates and the like are
25 contained as an antistatic agent and the sulfonates and the like contain minimum inorganic salts, for producing antistatic polyurethane elastic fiber.

Further object of the present invention is to provide antistatic polyurethane elastic fiber having
30 sufficient tenacity and elongation as an elastic fiber.

Further object and advantage of the present invention are clearly illustrated in the following description.

First, the above object and advantage of the present invention are achieved by a material for producing antistatic polyurethane elastic fiber comprising a mixture of 5 to 95 parts by weight of at least one salt selected from the group consisting of sulfonates having C_{8-30} hydrocarbon chain, sulfates having C_{8-30} hydrocarbon chain and phosphates having C_{8-50} hydrocarbon chain; and 95 to 5 parts by weight (based on 100 parts by weight of the total of the mixture) of a starting material other than organic isocyanate for producing polyurethane elastic fiber.

Secondly, the above object and advantage of the present invention are achieved by an antistatic polyurethane elastic fiber containing 0.1 to 10 weight percent of the above-mentioned salts and 0.1 to 10 weight percent of lubricants, and having a tenacity of 1 g/de or more and an elongation of 400 % or more.

Brief description of drawing

Figure 1 illustrates the device for measuring the yarn tension in a simulated knitting operation.

Preferred embodiment of invention

The material referred to in the present invention is a material for producing an antistatic polyurethane elastic fiber. The polyurethane elastic fiber of the antistatic polyurethane elastic fiber is the polyurethane fiber produced from the starting material comprising organic diisocyanates, long-chain glycols such as polytetramethylene glycol and polyesterdiol, and short-chain bifunctional compounds such as 1,2-propylenediamine and 1,4-butanediol. Such fiber can be produced by dissolving polyurethane in spinning solvent to prepare a dope and by spinning the dope in a well-

known manner.

The salts applied as an antistatic agent to the material of the present invention are sulfonates having C_{8-30} hydrocarbon chain, sulfates having C_{8-30} hydrocarbon chain and phosphates having C_{8-50} hydrocarbon chain. Either one or more of those salts can be used for the material.

Preferable sulfonates having C_{8-30} hydrocarbon chain are, for example, potassium alkanesulfonate having 15.5 carbon atoms on the average, lithium alkanesulfonate having 10.5 carbon atoms on the average, sodium dodecylbenzenesulfonate, sodium dibutyl-naphthalenesulfonate, tetrabutyl-phosphonium toluenesulfonate, trioctylmethyammonium toluenesulfonate, sodium polyoxyethylene lauryl ether propane sulfonate, potassium nonylphenyl ether propane sulfonate, sodium petroleum sulfonate and the like.

Preferable sulfates having C_{8-30} hydrocarbon chain are, for example, sodium octyl sulfate, potassium stearyl sulfate, tetrabutylphosphonium cetyl sulfate, sodium polyoxyethylene lauryl ether sulfate, potassium polyoxyethylene nonylphenyl ether sulfate, lithium castor oil sulfate, sodium sulfate methylricinoleate and the like.

Preferable phosphates having C_{8-50} hydrocarbon chain are, for example, sodium mono- and dilauryl phosphate, potassium mono- and distearyl phosphate, sodium mono- and dipolyoxyethylene lauryl ether phosphate, potassium mono- and dipolyoxyethylene nonylphenyl ether phosphate, and sodium mono- and dibutyl phosphate.

The said antistatic component of the present invention must be free from the groups reactive with organic isocyanates. And metal salts are preferable as the antistatic component for their antistatic effect.

According to the present invention, the polyurethane elastic fiber contains 0.1 to 10 weight percent, preferably 0.3 to 3 weight percent, of the said antistatic agent. The amount beyond the above range
5 results in insufficient antistatic effect or lowered tenacity and elongation.

The amount of inorganic matter in the antistatic agent is preferably 0.5 weight percent or less, more preferably 0.1 weight percent or less. Greater amount of
10 the inorganic matter causes fiber breakage and spinning pack choking.

The said material of the present invention contains salts such as the above-mentioned sulfonates and a starting material for producing polyurethane elastic
15 fiber other than organic diisocyanates. The preferable ratio of the former, the salts, is 5 to 95 parts by weight and that of the latter, the material, is 95 to 5 parts by weight based on 100 parts by weight of their total.

20 The latter, the starting material for producing polyurethane elastic fiber includes, for example, long-chain glycols and short-chain bifunctional compounds for producing polyurethane, spinning solvent, lubricants, antioxidants, and ultraviolet-ray absorbers. Either one
25 or more of those materials can be used. Long-chain glycols, spinning solvent, and lubricants are preferable among them.

The long-chain glycols for producing polyurethane include, for example, polytetramethylene glycol,
30 polyesterdiol, polypropylene glycol and polyethylene glycol. Among those compounds, polytetramethylene glycol and polyesterdiol are preferable.

The short-chain bifunctional compounds for producing polyurethane include, for example, succinic

acid, adipic acid, ethylene glycol, propylene glycol, 1,4-butanediol, hexanediol, hydrazine, 1,2-propylenediamine, 1,4-butylenediamine, 1,6-hexamethylenediamine, and m-xylylene-diamine.

5 The spinning solvent includes, for example, dimethylformamide, N,N'-dimethylacetamide, N,N,N',N'-tetramethylurea, N-methylpyrrolidone, and dimethyl sulfoxide. Among those, N,N-dimethylformamide and N,N-dimethylacetamide are preferable.

10 The lubricants include, for example, metal salts of saturated higher fatty acid such as magnesium stearate, modified silicones such as amino-modified silicone, alkylether-modified silicone and polyether-modified silicone, and higher fatty acid amide. Among those,
15 modified silicone and bisamide such as ethylenebisstearic acid amide are preferable.

 The antistatic polyurethane elastic fiber of the present invention can be produced advantageously from the above material of the present invention. The water
20 and alcohol contained in the above-mentioned material of the present invention can be decreased to a very low level before the material is mixed with organic diisocyanates through blending the material with a starting material for producing polyurethane elastic
25 fiber other than organic diisocyanates and drying the mixture in a well-known manner such as drying under low-pressure. The preferable amount of each water and alcohol in the material of the present invention is 0.5 weight percent or less, more preferably 0.1 weight
30 percent or less.

 The material of the present invention is processed into polyurethane elastic fiber in a well-known process where the material of the present invention is treated in the same manner as that for an ordinary starting

material for producing polyurethane elastic fiber other than organic diisocyanates.

The present invention provides an antistatic polyurethane elastic fiber containing 0.1 to 10 weight percent of the above-mentioned salt as an antistatic agent and 0.1 to 10 weight percent of a lubricant, and having a tenacity of 1 g/de or more and elongation of 400 % or more.

The fiber of the present invention can be produced from the said material of the present invention without decrease of the degree of polymerization, generation of oligomer, lowered tenacity and elongation of resultant fiber and generation of deposit, owing to the low amount of water, alcohol and inorganic salt in the material. In addition, the fiber of the present invention has uniform antistaticity because the antistatic component being dispersed in the material prior to fiber production easily mixes into spinning dope and disperses homogeneously.

Examples

The present invention is specifically explained with the following examples.

Example 1

Mono- and dipolyoxyethylene lauryl ether phosphate was neutralized with an aqueous solution containing 50-% potassium hydroxide and made into a polytetramethylene glycol solution containing 50 % of mono- and dipolyoxyethylene lauryl ether phosphate. The solution was heated up to 130°C and the water in the solution was evaporated at -700mmHg to obtain polytetramethylene glycol solution of potassium mono- and dipolyoxyethylene lauryl ether phosphate containing 200 ppm of water and 0.05 weight percent of inorganic salt

(hereinafter referred to as Additive a).

Example 2

Crude sodium octyl sulfate containing sodium sulfate decahydrate was dissolved in ethanol to precipitate sodium sulfate decahydrate and filtered. The filtered solution was dried to obtain a powder containing 0.05 weight percent of ethanol, 0.02 weight percent of water and 0.01 weight percent of inorganic salt. Then the powder was made into N,N-dimethyl acetoamide (DMAc) solution containing 10 weight percent of the powder (hereinafter referred to as Additive b).

Example 3

Crude dodecylbenzenesulfonic acid containing sulfuric acid was neutralized with a mixture of sodium hydroxide and methanol to precipitate sodium sulfate decahydrate and to obtain a methanolic solution containing 70 % of sodium dodecylbenzenesulfonate. Sixty seven parts by weight of polyether-modified silicone (EO:PO=60:40, viscosity 3000 cSt at 25°C) was added to 47 parts by weight of the solution, and the mixture was heated up to 130°C to evaporate methanol at -700mmHg and to obtain a solution containing 0.06 weight percent of water, 0.05 weight percent of methanol and 0.01 weight percent of inorganic salt (herein-after referred to as Additive c).

Example 4

Crude sodium alkanesulfonate having 15.5 carbon atoms on the average and containing sodium sulfate decahydrate was dissolved in methanol to precipitate sodium sulfate decahydrate and was filtered. After filtering the solution, ethylenebisstearic acid amide was added to the solution to 20 weight percent of the sodium alkanesulfonate having 15.5 carbon atoms on the average. Then the solution was heated up to 130°C and

vacuum dried at -700mmHg to obtain a solution containing 0.4 weight percent of water, 0.1 weight percent of methanol and 0.02 weight percent of inorganic salt (hereinafter referred to as Additive d).

5 Example 5

The mixture of 98 parts by weight of polytetramethylene glycol of 1500 number average molecular weight, 2 parts by weight of Additive a, and 33 parts by weight of 4,4-diphenylmethane diisocyanate was reacted at 70°C, and then 266 parts by weight of N,N-dimethylacetamide was added to dissolve the reacted mixture with cooling. After the solution was cooled down to 5°C, a solution prepared by dissolving 5 parts by weight of 1,2-diaminopropane in 184 parts by weight of N,N-dimethylacetamide, and 10 parts by weight of Additive a were added to the solution. The spinning dope prepared in the above procedure was fed to a spinneret having four fine holes, and extruded at 200 m/min into hot air in which solvent was evaporated. The extruded fiber was applied with 5 parts by weight of a 1 to 1 mixture of a dimethyl silicone having a viscosity of 10 cSt and a mineral oil of 60 sec, and wound up into 40 denier thickness. The properties of the resultant fiber are shown in Table 1.

25 Example 6

The mixture of 100 parts by weight of polytetramethylene glycol of 2000 number average molecular weight and 25 parts by weight of 4,4-diphenylmethane diisocyanate was reacted at 70°C. Then 250 parts by weight of N,N-dimethyl acetoamide was added to the reaction mixture to dissolve the mixture with cooling. After cooling down the solution at 5°C, a solution prepared by dissolving 3.7 parts by weight of 1,2-diaminopropane in 183 parts by weight of N,N-

dimethylacetamide and 10 parts by weight of Additive b were added to the solution. The spinning dope obtained in the above procedure was spun into fiber in the same manner as in Example 5. The properties of the resultant fiber are shown in Table 1.

Example 7

A fiber was spun in the same manner as in Example 6 except that 2 parts by weight of Additive c was added instead of 10 parts by weight of Additive b. The properties of the resultant fiber are shown in Table 1.

Example 8

The mixture of 100 parts by weight of polymethylpentanediol adipate of 2000 number average molecular weight, 9 parts by weight of 1,4-butanediol, 37.5 parts by weight of 4,4-diphenylmethane diisocyanate, and 2 parts by weight of Additive d was reacted at 85°C. The resultant reaction product was taken out of the kneader, and extruded at 200°C from four fine holes with an extruder at 200 m/min. The extruded fiber was applied with 5 parts by weight of a 1 to 1 mixture of a dimethyl silicone having a viscosity of 10 cSt and a mineral oil of 60 sec, and wound into 40 denier thickness. The properties of the resultant fiber are shown in Table 1.

Example 9

A fiber was spun in the same manner as in Example 8 except that the amount of Additive d was changed to 0.5 parts by weight. The properties of the resultant fiber are shown in Table 1.

Example 10

A fiber was spun in the same manner as in Example 6 except that 0.5 parts by weight of Additive c was added instead of 10 parts by weight of Additive b. The properties of the resultant fiber are shown in Table 1.

Comparative Example 1

A fiber was spun in the same manner as in Example 7 except that a commercially available alkanesulfonate having 14.5 carbon atoms on the average (containing 4 weight percent of inorganic salt and 2 weight percent of water) instead of Additive c. The pack pressure increased when the dope was extruded from the spinneret, and the dope could be extruded only for 5 hours. The resultant elastic yarn had an elongation of 270 % and a tenacity of 0.5 g/de, and was not used as elastic yarn.

Comparative Example 2

An elastic yarn was spun in the same manner as in Example 1 except that no additives were added. The properties of the resultant fiber are shown in Table 1.

Knitting tension:

The testing mechanism of a yarn in knitting operation is shown in Figure 1. A polyurethane yarn (2) unwound from a cheese (1) is driven through a compensator (3), rollers (4), knitting needles (5), a roller (7) attached to a U-gauge (6), and a speed meter (8), and connected to a winding roller (9). Yarn speed was controlled constant at a given speed (for example, 10 m/min or 100 m/min) with the speed meter (8), and a yarn was wound onto the winding roll. The variation of the yarn tension while the winding operation was measured with the U-gauge (6) to determine the friction (g) between the yarn and the knitting needles.

Static charge:

An electrometer (KS-525, Kasuga Electric Company) was placed 1 cm above the U-gauge, and the static charge on the driven yarn was detected.

Specific resistance:

The specific resistance was tested with a Fiber Tester (Type MR-2010, Dempa Ind. Co., Ltd.).

Table 1

Test No.	Ex. 5	Ex. 6	Ex. 7	Ex. 8	Ex. 9	Ex. 10	Comp. Ex. 2
Knitting tension (g)							
10 m/min	20	21	20	20	21	20	20
100 m/min	25	27	23	25	25	24	25
Static charge(kv)	0.1	0.1	0.1	0.2	0.3	0.1	0.5
10 m/min	0.2	0.2	0.1	0.3	0.3	0.2	2.0
100 m/min							
Specific resistance($\Omega \cdot \text{cm}$)	2×10^8	3×10^8	1×10^8	4×10^8	5×10^8	2×10^8	4×10^{11}
Tenacity (g/de)	2.3	2.6	2.4	2.3	2.0	2.4	2.3
Elongation (%)	590	620	610	560	570	610	580

Ex.: Example

Comp. Ex.: Comparative Example

- 5 The material of the present invention enables to produce yarn having superior properties (elongation of 400 % or more and tenacity of 1 g/de or more), to prevent static charge on elastic yarn in covering and beaming process, and to decrease troubles such as yarn
- 10 breakage.

CLAIMS

1. A material for producing antistatic polyurethane elastic fiber, said material being the mixture (of which
5 total is 100 parts by weight) of 5 to 95 parts by weight of at least one salt selected from the group consisting of sulfonates having C₈₋₃₀ hydrocarbon chain, sulfates having C₈₋₃₀ hydrocarbon chain and phosphates having C₈₋₅₀ hydrocarbon chain, and 95 to 5 parts by weight of a
10 starting material for producing polyurethane elastic fiber other than organic isocyanate.

2. A material in Claim 1, wherein the starting material for producing polyurethane elastic fiber is
15 selected from the group consisting of long-chain glycol for producing polyurethane, spinning solvent, and lubricants.

3. A material in Claim 1, wherein the long-chain
20 glycol for producing polyurethane elastic fiber is polytetramethylene glycol or polyesterdiol.

4. A material in Claim 1, wherein the spinning solvent is N,N-dimethyl-formamide or N,N-dimethylacetamide.
25

5. A material in Claim 1, wherein the lubricants are bisamides or modified silicones.

6. A material in Claim 1, wherein the said salts
30 contain 0.5 weight percent or less of inorganic salts.

7. (after amendment) An antistatic polyurethane elastic fiber containing 0.1 to 10 weight percent of at least one salt selected from the group consisting of

sulfonates having C_{8-30} hydrocarbon chain, sulfates having C_{8-30} hydrocarbon chain and phosphates having C_{8-50} hydrocarbon chain (in which 0.5 weight percent or less of inorganic salts to the weight of the metal salts are contained), and 0.1 to 10 weight percent of the lubricants, and having a tenacity of 1 g/de or more and an elongation of 400 % or more.

ABSTRACT

Inorganic salts are generated in the production process of metal salts, such as sulfonates, sulfates and phosphates. When such metal salts containing inorganic salts are added as an antistatic agent to a polymer for producing polyurethane fiber, the inorganic salts cause fiber breakage or pack choking in fiber extrusion process. In addition, such antistatic agents are highly hygroscopic and contain trace of water. When such an antistatic agent is added to the material for polymerizing polyurethane, the alcohol and water in the agent react with isocyanate to result in lowered degree of polymerization and generation of oligomer. Such polyurethane polymer is spun into fiber of low elongation and tenacity. The material for polyurethane elastic fibers of the present invention comprises a mixture of 5 to 95 weight percent of at least one of the above-mentioned antistatic agents and 95 to 5 weight percent of a isocyanate-free starting material for elastic fibers, and eliminates the above troubles.

Dkt. No. OHS-285**DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION**

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (*if only one name is listed below*) or an original, first and joint inventor (*if plural names are listed below*) of the subject matter which is claimed and for which a patent is sought on the invention entitled
ANTISTATIC POLYURETHANE ELASTIC FIBER AND MATERIAL FOR PRODUCING THE SAME

_____, the specification of which:

(check one) ☐ is attached hereto ☒ was filed on May 19, 2000 as
Application Serial No. 09/554,736 and was amended on _____ (*if applicable*).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR §1.56.

I hereby claim foreign priority benefits under 35 U.S.C. §119(a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate, or §365(a) of any PCT international application which designated at least one country other than the United States, listed below and have also identified below any foreign application for patent or inventor's certificate, or PCT international application having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s)

10-014890

(NUMBER)

Japan

(COUNTRY)

09/01/1998

(FILED D/M/Y)

Priority Claimed

☒ ☐

YES NO

88100261

(NUMBER)

Taiwan

(COUNTRY)

08/01/1999

(FILED D/M/Y)

☐ ☒

YES NO

(NUMBER)

(COUNTRY)

(FILED D/M/Y)

☐ ☐

YES NO

(NUMBER)

(COUNTRY)

(FILED D/M/Y)

☐ ☐

YES NO

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YES NO

(NUMBER)

(COUNTRY)

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(FILED D/M/Y)

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YES NO

I hereby claim the benefit under 35 U.S.C. §120 of any United States application(s), or §365(c) of any PCT international application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT international application in the manner provided by the first paragraph of 35 U.S.C. §112, I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

PCT/JP99/00029
(APPLICATION SERIAL NO.)January 8, 1999
(FILING DATE)Pending
(STATUS)

(APPLICATION SERIAL NO.)

(FILING DATE)

(STATUS)

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine and imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or document or any patent issuing thereon.

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☐ ADDITIONAL INVENTORS ARE BEING NAMED ON SEPARATELY NUMBERED SHEETS ATTACHED HERETO